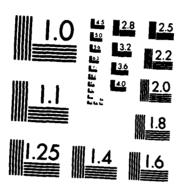
IR PHOTOACOUSTIC STUDIES OF SURFACE REACTIONS(U) NEW-YORK UNIV NY DEPT OF CHEMISTRY M J LOW 20 MAY 83 8 ARO-16631 18-CH DARD29-79-C-0135 UNCLASSIFIED F/G 7/4 NL

1/1

AD-A129 778



MICROCOPY RESOLUTION TEST CHART
NATIONAL BUREAU OF STANDARDS-1963-A



Unclassified
SECURITY CLASSIFICATION OF THIS PAGE (When Date Entered)

ARO 16631.18-CH

REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER 8	2. JOYT ACCESSION NO.	3. RECIPIENT'S CATALOG NUMBER
IR PHOTOACOUSTIC STUDIES OF SURFACE REACTIONS		5. TYPE OF REPORT & PERIOD COVERED Final Report 7/1/79 - 3/31/83
		6. PERFORMING ORG. REPORT NUMBER
7. AUTHOR(a)		B. CONTRACT OR GRANT NUMBER(#)
M.J.D.Iow		DAAD 29-79-C-0135
9. PERFORMING ORGANIZATION NAME AND ADDRESS		IO. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS
Department of Chemistry		·
4 Washington Place		na
New York, N.Y. 10003		
U. S. Army Research Office		12. REPORT, BATE 5/20/83
P. O. Box 12211		13. NUMBER OF PAGES
Research Triangle Park, 30 27709		<u> </u>
14. MONITORING AGENCY NAME & ADDRESS(II differen	t from Controlling Office)	15. SECURITY CLASS. (of this report)
Department of the Navy Office of Naval Research Resident Representative 715 Broadway (5th Floor) New York,NY 10003		Unclassified
		15. DECLASSIFICATION/DOWNGRADING

16. DISTRIBUTION STATEMENT (of this Report)

Approved for public release; distribution unlimited.

SELECTE JUN 2 3 1983

17. DISTRIBUTION STATEMENT (of the obstract entered in Block 20, if different from Report)

B

18. SUPPLEMENTARY NOTES

The view, spinions, and/or findings contained in this report are those of the author(s) and should not be construed as an official Department of the Army position, policy, or decision, unless so designated by other documentation.

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)

Infrared spectroscopy
Photoacoustic spectroscopy
Photothermal spectroscopy
Surface reactions
Surface species

20. ABSTRACT (Continue on reverse elde il necessary and identify by block number)

An infrared Fourier transform photothermal beam deflection spectrometer system was constructed and used to record spectra of solids. No sample preparation is needed. The surfaces of massive objects can be studied. The techniques are applicable to surface studies in which samples must be examined under rigorously controlled conditions. Details are given in 20 publications, which are listed.

83 06 22 061

FILE CO

DD FORM 1473 EDITION OF I NOV 65 IS OBSOLETE

### FINAL REPORT

# PERFORMANCE PERIOD

The original performance period was 1 July, 1979 to 30 June, 1982. In order to permit some work to come to a conclusion and to permit papers to be published, the period was extended without additional funds to 30 June, 1983 but then the termination was modified to 31 March 1983. As almost no funds remained past June 1982, little new work was undertaken.

# SUMMARY OF WORK

The work which has been carried out has been briefly outlined in seven Progress Reports issued at intervals of six months, but has already been described in detail in twenty research publications; a list of these is given below. The work can be summarized as follows.

The main aim of the work was to develop the techniques of photoacoustic spectroscopy (PAS), specifically in the infrared (IR) range, in order to apply IR/PAS to the study of surfaces of samples which are rough and opaque.

Work was begun with the construction and development of dispersive IR/PAS spectrometers, and the successful application of the instruments to the study of solids and surface species on the surfaces of solids was shown. This work is fully described in publications 1 through 7 and 10. Simultaneously, work was begun on the construction of a Fourier transform (FT) IR/PAS instrument but, before it could be made operational, the effort was anticipated in the literature. Work on IR/FT/PAS was stopped. however, for another reason : the discovery of a new detection method. Boccara, Fournier and Badoz ( Appl.Phys.Lett. 36,130 (1980) ) described a new technique by which the photoacoustic effect could be detected by observing the deflection of a light beam passing over the sample's surface; the thermally-induced refractive index changes over the sample cause a light beam to deflect. By measuring the extent of the beam deflection with a position-sensing detector, a measure of the extent of the photothermal effect can be obtained. This new detection method seemed to offer many advantages with respect to PAS, so work with IR/PAS was abandoned and directed toward the development of IR/FT photothermal beam deflection spectroscopy (PBDS).

Extensive effort was required to make IR/FT/PBDS "work" in the IR range, but a spectrometer system was successfully completed and a variety of techniques were developed. This work is fully described in publications Y and 11 through 20.

IR/FT/PBDS has been shown to be highly successful for the qualitative study of solids and the surfaces of solids. The advantages of the technique are :

1. No sample preparation whatsoever is required. Tedious. time-

consuming and costly sample manipulations usually required for the infrared examination of solids are not needed, and potential changes in sample composition or structure induced by sample preparation steps are avoided.

- 2. Massive solid objects may be examined, without removing a sample from the object. In order to examine a solid, all that is required is to position the object so that the IR and probe laser beams fall on and pass over a 2 mm area on the object's surface (however, an area within a depression on the surface cannot be studied). Consequently, all size limitations imposed on samples by conventional IR techniques and by IR/PAS are removed. It was presently convenient to construct the instrumentation so that selected areas on the surfaces of objects about 10 x 10 x 10 cm could be examined. However, the instrument can be re-configured so that any size object could be examined.
- 3. Sample cells per se are not needed, in view of item 2.
- 4. If a sample must be confined within a cell or enclosure in order to protect it from contamination or to expose it to noxious materials, this may be done with ease, even with large objects such as entire catalyst pellets.
- 5. If a sample must be confined, as must be done for surface studies, the sample can be studied under the rigorously clean conditions normal to surface studies. The detection system is outside of the sample cell, unlike the case of PAS, so that the sample cannot be contaminated by gases evolved by the detector.
- 6. Most samples which are optically opaque can be examined. The IR/FT/PBDS techniques have been used to study the surface properties of carbons as part of another project, with excellent results ( 3 papers are in press with the journal Carbon).

Although IR/FT/PBDS has been shown to be an excellent qualitative technique, at present the following difficulties arise:

- A. Placing a sample correctly in position is difficult; the intensity of the signal obtained is crucially dependent on the relative positions of the sample surface, IR beam, and laser probe beam. Consequently, at present the quantitative aspects of the technique are poor. It is expected that future work will lead to significant improvements.
- B. As the technique depends on sensing changes in beam position occurring in the 400-2000 Hz range, mechanical vibrations and airborne sound falling in that frequency range cause noise, so that the equipment must be properly sound-proofed and shielded.

### **PUBLICATIONS**

- 1. N.Madison and M.J.D.Low, A bolt-on step drive for monochromators. Chem., Biomed., and Environ. Instrumentation 10, 209(1980).
- 2. M.J.D.Low and G.A.Parodi, Infrared photoacoustic spectra of solids. Spectrosc.Lett. 13, 151 (1980).
- 3.M.J.D.Low and G.A.Parodi, Infrared photoacoustic spectroscopy of surfaces, J. Molec. Struct. 61, 119 (1980).
- 4.M.J.D.Low and G.A.Parodi, An infrared photoacoustic spectrometer, Infrared Phys. 20, 333(1980).
- 5.M.J.D.Low and G.A.Parodi, Infrared photoacoustic spectroscopy of solids and surface species, Appl. Spectrosc. 34,76(1980).
- 6.M.J.D.Low and G.A.Parodi, Carbon as reference for photoacoustic spectra, Spectrosc.Lett.13,663(1980).
- 7.M.J.D.Low and G.Parodi, A photoacoustic cell for use with solids, Chem. Biomed. Environm. Instrum. 10, 397 (1980)
- 8. M.J.D.Low, Microgravimetric/Infrared Studies: a review. J.Vac Sci.Technol. 17,98(1980).
- 9.M.J.D.Low, G.A.Parodi and M.Lacroix, A detection system for photoacoustic spectroscopy. Chem., Biomed. Environm. Instrum. 11, 265(1981).
- 10.M.J.D.Low and G.A.Parodi, Dispersive photoacoustic spectroscopy of solids in the infrared range, J.Photoacoust. 1, 131 (1982).
- 11. M.J.D.Low and M.Lacroix, An infrared photothermal beam deflection Fourier transform spectrometer, Infrared Phys. 22, 139(1982).
- 12.M.J.D.Low, C.Morterra and A.G.Severdia, Infrared photothermal deflection spectroscopy of carbon-supported metal catalysts, Spectrosc.Lett.15,415(1982).
- 13.M.J.D.Low, C.Morterra and M.Lacroix, Destruction of solid samples by photoacoustic spectroscopy, Spectrosc.Lett.15, 159(1982).
- 14.M.J.D.Low, M.Lacroix and C.Morterra, Infrared Spectra of massive solids by photoacoustic beam deflection Fourier transform spectroscopy, Spectrosc.Lett.15,57(1982).
- 15.M.J.D.Low, M.Lacroix, C.Morterra and A.G.Severdia, Infrared spectra of solids and large objects, American Laboratory, June 1982, p 16.

16.C.Morterra, M.J.D.Low and A.G.Severdia, Some effects of specular and diffuse reflection on infrared photoacoustic spectra. Infrared Phys. 22,271(1982).

17.M.J.D.Low, C.Morterra and A.G.Severdia, Errors and artifacts in infrared bands of CO chemisorbed on supported metals. Spectrosc. Lett. 15,565(1982).

18.M.J.D.Low, M.Lacroix and C.Morterra, Infrared photothermal beam deflection Fourier transform spectroscopy of solids, Appl. Spectrosc. 36,582(1982).

19.M.J.D.Low, C.Morterra, A.G. Severdia and M.Lacroix, Infrared photothermal deflection spectroscopy for the study of surfaces, Appl. Surface Sci. 13, 429(1982).

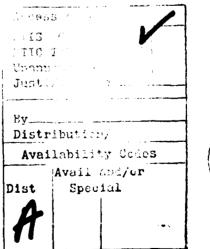
20. M.J.D.Low, A.G.Severdia and T.H.Arnold, The nature of the coupling gas and its pressure on photothermal beam deflection spectra. Infrared Physics, in press.

# SCIENTIFIC PERSONNEL SUPPORTED BY THIS PROJECT

M.J.D.Low , Principal Investigator (part time)

G.A.Parodi , M.Lacroix, C.Morterra , A.G.Severdia , T.H.Arnold, Assistant Research Scientists , part time.

No degrees were awarded.





# FILMED

7-83